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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : C09K 3/30, B01J 13/00 A61L 9/04, A01N 29/00	A1	(11) International Publication Number: WO 92/05229 (43) International Publication Date: 2 April 1992 (02.04.92)
(21) International Application Number: PCT/US91/06635 (22) International Filing Date: 13 September 1991 (13.09.91) (30) Priority data: 584,963 19 September 1990 (19.09.90) US (71) Applicant: S.C. JOHNSON & SON, INC. [US/US]; Patent Section, MS 077, 1525 Howe Street, Racine, WI 53403-5011 (US). (72) Inventor: NEUMILLER, Phillip, J. ; 10017 Spring Street, Racine, WI 53406 (US). (74) Agents: SANDER, Dorothy, L. et al.; Patent Section MS 077, S. C. Johnson & Son, Inc., 1525 Howe Street, Racine, WI 53403-5011 (US).		(81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), SE (European patent). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: AQUEOUS EMULSION AND AEROSOL DELIVERY SYSTEM USING SAME (57) Abstract <p>An aqueous emulsion system containing vesicular structures which is used to prepare an aerosol delivery system. The vesicular structures of the emulsion provide a reservoiring effect for the propellant, enabling the aerosol system to be effective with a very low VOC content.</p> <div data-bbox="889 1186 1347 1837"><p style="text-align: right;">1 μm 100 nm</p></div>		

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Aqueous Emulsion and Aerosol Delivery System Using Same
Technical Field

This invention relates to the field of aqueous emulsion
5 systems and the use of such systems for dispensing aerosols
from pressurized containers and more particularly to an
improved emulsion system which contains vesicle structures
that can be used to provide a reservoiring effect for the
propellant component of an aerosol delivery system.

10 Background Art

A vesicular system may be considered as a particular
type of emulsion system, in which the dispersed or emulsified
phase particles are layered vesicles which are suspended in
the continuous phase.

15 The fact that vesicular systems can be formed and then
used to entrap and carry desirable active compounds is well
known. Such systems have most frequently been formed from
organic materials of biological origin such as lipids (see,
e.g., U.S. Pat. No. 4,772,471), Vitamin E (U.S. Pat.
20 No. 4,861,580), or steroids (U.S. Pat. No. 4,917,951), and
have been especially used in the pharmaceutical fields to
provide carriers for biologically active materials.

A method of making, from non-phospho-lipid surfactants,
paucilamellar vesicles having a central cavity substantially
25 filled with a water-immiscible oily material is disclosed by
U.S. Pat. No. 4,911,928 to Wallach, Paucilamellar Lipid
Vesicles. The "lipid vesicles" disclosed by this patent are
large (500 nm diameter minimum) multilayered liposome-like
structures which are centrifuged (at 10,000 - 14,000 rpm for
30 15 minutes) out of the system after formation.

The creation of a vesicular dispersion from non-ionic
surfactants is disclosed by U.S. Pat. No. 4,536,324 to
Fujiwara et al., Nonionic Surfactant Type Vesicle Dispersion,
which discloses a vesicle system formed from non-ionic
35 surfactants such as polyoxethylene castor oil ethers or
hardened castor oil ethers combined with sorbitan polyesters
of long chain fatty acids in water. Conventional mixing
means, from mechanical to ultrasonic, are used to form the
vesicle dispersion or emulsion. Suggested uses for the

dispersion or emulsion are either alone as a cosmetic cream or lotion or for containing a lipophilic or hydrophilic pharmaceutically active component.

The creation of a vesicle system from a mixture of cationic and anionic surfactants in water has been reported by Kaler et al. (Science, Sept. 22, 1989, p. 1371) Gentle mixing of cetyltrimethyl ammonium tosylate and sodium dodecyl benzene resulted in immediate and spontaneous (no mechanical agitation) generation of vesicles having particle sizes between 30 and 80 nm. The vesicles so formed were said to be stable and able to efficiently encapsulate glucose or other solutes.

Many two component systems for the delivery of an aerosol from a pressurized container are known. One component of such a system must be a gaseous propellant. The other component is a liquid component, which contains the active ingredient to be dispersed, may be comprised of various solvents, or may have an aqueous base with added solvents.

Many propellants currently used (since the use of nonflammable chlorinated fluorocarbons is being limited for environmental reasons) are flammable hydrocarbon gases such as propane, butane, and isobutane. Aqueous systems are preferred for use with such propellants, since such a system can limit or even obviate the flammability of the propellant phase.

The use of microemulsions in an aerosol system is known. U.S. Pat. No. 4,655,959, Preparation of Non-Flammable Aerosol Propellant Microemulsion System, and 4,536,323, Non-Flammable Propellant Microemulsion System, both issued to Stopper, disclose an aerosol system which, when shaken in its container, forms an oil-in-water microemulsion. This microemulsion structure allows the amount of propellant to be increased up to 50% by weight without flammability problems. Stopper's reason for wishing to have a higher level of propellant than the 15% to 25% conventional limit for non-flammability is his desire to be able to disperse the entire contents of the dispenser.

A countervailing consideration to the desire to incorporate a large amount of propellant into an aerosol system for efficiency of delivery is the desire to limit the amount of volatile organic compounds (VOCs) released into the earth's atmosphere. More immediately critical is the need to reduce the amount of VOCs into the home, for there is concern that indoor air pollution may sometimes exceed external air pollution and this is becoming an issue as a potentially health affecting condition. California, for example, is developing maximum VOC concentration regulations for different product categories. The proposed limit for air freshener double phase aerosols is 30%; for insect repellents, it is 65%. Analysis of one of the lowest VOC-containing aerosols currently on the market shows a VOC content of 28%.

An article in Aerosol Age ("CARB/Industry Negotiate Consumer Product Regs.", July 1990, pp. 22-27) has a table showing the differences between the proposed limits and "industry's needs". Industry need for air fresheners and disinfectants are said to be 70%; for dusting aids, 35%; for hair sprays, 80%. The present invention appears to offer a system that can deliver a variety of active ingredients, making it useable for a wide variety of products, with a total VOC content far below the "industry needs".

Which compounds qualify as VOCs will depend on type as well as molecular weight but, in general, organic compounds with fewer than nine carbon atoms are usually considered potential VOCs. Propane, butane, and isobutane are, obviously, VOCs.

Thus a non-flammable aqueous aerosol system that could effectively deliver the container contents with a lower, rather than a higher, level of propellant is highly desirable both for environmental and regulatory reasons. However, prior art has not only not produced such a system but, as in the Stopper patents, even teaches away from such a possibility.

All aerosol systems require a certain minimum propellant head space pressure to expel the contents of the container. Propellant head space pressure is dependent upon the

interaction the propellant has with other substances in the container.

A container charged with propane alone (no other substances in the container) will exhibit a head space pressure of 100-110 psi (7031 gr/sq. cm - 7734.1 gr/sq.cm). A container containing water that is then filled with propane will exhibit a head space pressure of 110-120 psi (7834.1 gr/sq. cm - 8437.2 gr/sq. cm). When alcohol, glycerol, or a surfactant is added to the water, the head space pressure can be lowered. A mixture of 66% water, 30% ethanol, and 4% propane will exhibit a head space pressure of 50 psi, which is a near optimum head space pressure for an aerosol system which will produce a spray. 55 psi (3867 gr/sq. cm) pressure is considered the optimum figure.

A further consideration for an effective aerosol system is the ability of the system to maintain the desired pressure as the contents and the propellant are expelled. The alcohol-water-propane system described above exhibits progressively decreased head space pressure as the contents are expelled from an aerosol container with a vapor tap valve.

A desirable aerosol system should thus have the capacity to entrap or reservoir some of the propellant phase and progressively release the propellant as the contents of the aerosol container are expelled, thus maintaining a constant-equilibrium head space pressure over most of the usable life of the aerosol container.

Summary Disclosure of the Invention

The present invention is a unique aqueous emulsion preparation which can be used for delivering an aerosol composition from a pressurized container. It is effective in dispensing the entire contents of the container, yet does so with a lower level of propellant and VOCs than previously possible. Total VOC's of the present invention range from 2% to 25%, compared to the current average aerosol VOC levels of 28% to 98%.

This is accomplished by the production, in the liquid component of the system, of an emulsion preparation that includes discrete vesicular particles which are suspended in

a continuous phase and kept in stable suspension by the mixture of surfactant, primary alcohol, and polyhydroxy alcohol or polyhydroxy alcohol ester chosen. The characterization of the discrete particles of the emulsion preparation as vesicles of an average size of 20-100 nm is indicated by photon correlation spectroscopy and confirmed by electron microscopy.

It has been found that such an emulsion preparation can be produced from a combination of non-ionic single- or double-tailed surfactants, primary alcohol, polyhydroxy alcohol or polyhydroxy alcohol ester, and an organic, preferably active, ingredient, and that such a system can entrap or provide a reservoiring effect for small linear organic propellant molecules in the C₃-C₅ range. The reservoiring effect resulting from increased partitioning of the propellant in these formulations due to enhanced mutual compatibility reduces the amount and/or the rate of loss of propellant into the gas phase when the formulation is exposed to atmospheric pressure by the opening of the vapor tap. This reservoiring effect functions similarly to an increased solubility - more propellant is held within the system and gradually released. This reservoiring equilibrium effect functions to regulate head space pressure within the pressurized container and prevents the decrease of head space pressure that would otherwise occur when a vapor tap valve is used. The fact that the propellant is reservoiried within the aqueous phase makes it possible for the system to function properly over the useful life of the aerosol container with a lower concentration of propellant rather than a higher one, which might logically be expected.

Best Mode for Carrying Out the Invention

The first step in the preparation of an aqueous aerosol delivery system according to the present invention is the preparation of an aqueous emulsion stage, which is the same as the aqueous component used to produce the aerosol delivery system.

A non-ionic surfactant or a mixture of non-ionic surfactants is mixed with a polyhydroxy alcohol or polyhydroxy alcohol ester and a primary alcohol. A

preservative or antimicrobial agent may be added. Then water is added and the mixture homogenized to form a lamellar or liquid crystal phase which is thick (viscosity 20-100 cS), translucent and often exhibits iridescence. An active
5 organic compound, such as a fragrance or an insecticide, is then added to the system. Part of the lamellar liquid crystal structure could be converted into multilamellar liposomes. The degree of this conversion depends mainly on the intensity of shear used in homogenization step. Measured
10 rheological properties such as significant elasticity, shear thinning behavior and high viscosity values at low shear rates indicate that the liquid crystal form is suitable for making shampoos, dermal, and cleaning or polishing formulations.

15 Next the lamellar stage is subjected to sonification, high energy shearing, or other type of energy addition. This produces a stable aqueous emulsion stage with lower viscosity (approximately 10 cS or lower) which is unclouded and transparent.

20 Three representative formulations of the invention (one of the lamellar or liquid crystal system and two of the vesicular system) were centrifuged for five hours at 17,000 rpm (102,000 Deg/Sec) (at an acceleration of 34,800 g) in a Sorvall Superspeed RC2B centrifuge using an SS34 rotor.
25 None of the formulations showed any phase separation. This behavior indicates a very high stability for both the lamellar and the vesicular systems, making them suitable for producing products with long shelf life.

Figures 1-3 all show representative TEM photographs
30 selected from over forty sets of photographs from four different formulations. All photographs showed, with expected minor variations, the same structural makeup of the emulsion and vesicular phases of the invention. All samples were rapidly quenched (frozen) to avoid formation of ice
35 crystals. Then the frozen sample was brought to vacuum and fractured. The samples were shadowed with platinum and photographed by TEM. Original TEM photographs were taken as stereo pairs, to be viewed with a stereoscope for maximum resolution.

Figure 1, taken at a magnification of 40,300, shows the multilamellar liposome structures contained in a representative preparation of this aqueous emulsion.

Figure 2, taken at a magnification of 62,000, shows the vesicular structures, which average 20-100 nm in diameter, present in the aqueous emulsion after it has been subjected to sonification.

Figure 3, taken of a slightly different but also representative formulation of the present invention and taken at a magnification of 46,500 shows that, while the majority of the vesicular structures present in the sonicated aqueous emulsion are between 20 and 100 nm, there are some larger unilamellar vesicular structures present.

According to one aspect of the present invention, the aqueous emulsion stage may be used to form delivery systems for such preparations as polishes, air fresheners, insecticides, cleaning products, dermal treatments, etc.

However, according to a further aspect of the present invention, the aqueous emulsion stage component is next placed into a pressurizable container, which is then charged with a propellant.

The aqueous aerosol delivery system is comprised of the aqueous emulsion stage component, which is present in between 75% to 98% by weight of the system, and a propellant component, present in between 2% to 25% by weight of the system.

The surfactants used to form the aqueous component of the present invention are non-ionic surfactants, which may either be of a single type having double hydrocarbon tails extending from the functional group or be a pair combination of two different types of surfactants having single hydrocarbon tails extending from their functional groups. Mixtures of such types of surfactants may also be used. Possible "double-tailed" non-ionic surfactants which may be used in the system of the present invention are the fatty acid alkanolamides, ethylene oxide adducts of the higher primary alcohols or an ethoxylated amines. Possible "single-tailed" surfactants, which must be used in pairs are sorbitan monooleate, polyoxyethylene (2) oleyl ether, and

polyoxyethylene (20) sorbitan monooleate. While it is usually desirable to use either a double-tailed surfactant or a pair combination of single-tailed surfactants, it is also possible to use both a double-tailed surfactant and a single-tailed surfactant combination. Such a combination reduces necessary total level of surfactant as well as providing an opportunity to alter the characteristics of the resulting systems for specific desired features. The surfactants are present in the liquid component in concentrations between 0.25% and 6.5%.

The primary alcohols used to form the aqueous component of the present invention range from ethanol to oleyl alcohol. It appears that a small quantity of a primary alcohol is essential to produce the reservoiring effect which characterizes the invention. It is theorized that this reservoiring effect is produced by the coupling of the propellant into the membranes of the vesicles.

Alcohols below C₉, however, are themselves volatile organic compounds, so the preferred alcohols of the present invention are linear C₉-C₁₈ (nonyl to oleyl) alcohols, with the most preferred alcohols being the C₁₀ (decanol) and C₁₁ (1-undecanol) alcohols. The primary alcohol is present in the aqueous component in concentrations between 0.001% and 3.5%.

The polyhydroxy alcohol or polyhydroxy alcohol ester used to form the aqueous emulsion stage component of the present invention is preferably a C₂-C₆ alcohol compound such as glycerol, ethylene glycol, or diethylene glycol. The polyhydroxy alcohol esters are preferably C₁₀-C₃₀ polyhydroxy alcohol esters. Mixtures of polyhydroxy alcohols and polyhydroxy alcohol esters may be used. Polyhydroxy alcohol ethers may also be useable. The polyhydroxy alcohol or polyhydroxy alcohol ester is present in the aqueous component at concentrations between 0.1% and 6%.

The aqueous component of the present invention may also include a preservative such as methylparaben, present at concentrations between 0.1% and 0.5%.

Included in the aqueous emulsion phase component of the present invention is an organic active ingredient chosen

according to the desired characteristics of the final product. Possible organic active ingredients could include fragrances, pesticides (such as pyrethrin or linalool) or repellents (including personal insect repellents such as N,
5 N-diethylamine-meta-toluamide (DEET)), waxes (including silicone oils), emollients, cleansers, etc. The active ingredient may be either lipophilic or hydrophilic. The organic active ingredient is present in the aqueous component at concentrations between 0.01% and 20%.

10 Water makes up the balance of the aqueous component for all formulations. It is preferred that deionized water be used.

The propellant component of the present invention is a linear chain hydrocarbon, such as propane, butane, pentane or
15 mixtures thereof. The propellant component is, as discussed above, present at concentrations between 2% and 25% by weight of the total system, and preferably between 2% and 10%.

The reservoiring effect of the aerosol system and the need for a long chain alcohol to produce that effect of the
20 present invention is best illustrated by the behavior of the system with two different propellant gases: isobutane and propane. Propane alone exhibits a head space pressure of 110 psi. When a non-ionic surfactant, such as a fatty acid alkanolamide, is added to the container, propane exhibits a
25 pressure of 96 psi (6749.76 gr/sq. cm). When propane is used as the propellant in the system of the present invention prepared without a long chain alcohol, the system has a pressure of 100 psi (7031 gr/sq. cm). When a long chain alcohol is present in the system, propane pressure is 55 psi,
30 (3867 gr/sq. cm) showing the coupling effect of the alcohol in the system with the propellant.

Isobutane alone exhibits a head space pressure of 35 psi (2460.85 gr/sq. cm). When a non-ionic surfactant, such as a fatty acid alkanolamide, is added to the container, the
35 pressure is 32 psi (2249.92 gr/sq. cm). When isobutane is used as the propellant in the system of the present invention (with a long chain primary alcohol present), the system has a pressure of 39 psi (2742 gr/sq. cm). Isobutane thus appears

unable to couple into the system even in the presence of a long chain primary alcohol.

It is believed that the reservoiring effect is produced by the penetration into or coupling with the membranes of the vesicular structures of the propellant molecules. Propane (as well as butane and n-pentane) molecules, being sterically slender, fit between the molecules of the membrane. Isobutane, however, being sterically more bulky, is unable to fit completely into the vesicular structure in this manner.

Combinations of non-ionic surfactants, primary alcohols, and polyhydroxy alcohols or polyhydroxy alcohol esters that have been tested and have produced stable aerosol systems use components selected from the following groups:

Preferred Surfactants

Single Surfactants (% concentration range 0.25 - 6.5):

- Fatty acid alkanolamide (Monamid 150 ADY)
- Linoleamide (Monamid B-442)
- Tallow monoethanolamide ethoxylate (Sherex T-55)
- Ethylene oxide adducts of nonylphenol (Surfonic N-85, Surfonic N-95, Surfonic N-100)

Surfactant Pairs

	Surfactant 1 (% concentration range)	Surfactant 2 (% concentrations)
25	Sorbitan monooleate (Span 80; 0.5 - 5.8)	Polyoxyethylene (2) oleyl ether (Tween 80; 0.1 - 5.3)
	Polyoxyethylene (2) oleyl ether (Brij 92; 0.5 - 6.0)	Polyoxyethylene (20) sorbitan monooleate (Tween 80; 0.1 - 5.3)
30	C ₉ -C ₁₁ linear alcohol ethoxylate (Neodol 91-2.5; 0.5 - 6.0)	Polyoxyethylene (20) sorbitan monooleate (Tween 80; 0.1 - 5.3)
	Block copolymer of propylene and ethylene oxide (Pluronic L-64; 0.3 - 5.8)	Sorbitan monooleate (Span 80; 0.1 - 5.2)
35	Fatty acid alkanolamide (Monamid 150 ADY; 0.5 - 6.5)	Octylphenoxy polyethoxyethanol (Triton X-35; 0.2 - 5.5)
	Glyceryl laurate (Kessco 675; 0.3 - 5.8)	Sorbitan monooleate (Span 80; 0.1 - 5.2)
40	Linoleamide (Monamid B-442; 0.5 - 6.0)	Polyoxyethylene (2) oleyl ether (Tween 80; 0.1 - 5.2)

Preferred Primary Alcohols (% concentration range 0.1 - 3.1)

- CH₃-(CH₂)₁₀-OH
Mixed C₉/C₁₀/C₁₁ alcohol (Neodol 91)
C₁₁ alcohol (Neodol 1)
5 CH₃-(CH₂)₁₁-OH
Mixed C₁₂/C₁₃ alcohol (Neodol 23)
CH₃-(CH₂)₁₃-OH
CH₃-(CH₂)₁₄-OH
Mixed C₁₄/C₁₅ alcohol (Neodol 45)
10 CH₃-(CH₂)₁₅-OH
CH₃-(CH₂)₁₆-OH
CH₃-(CH₂)₁₇-OH, Oleyl

Preferred Polyhydroxy Alcohols and Polyhydroxy Alcohol Esters

- 15 (% concentration range 0.1 - 6.0)
Glycerin, C₃H₅(OH)₃
Ethylene glycol, CH₂OHCH₂OH
1,2-Propylene glycol, CH₃CHOHCH₂OH
Diethylene glycol, CH₂OHCH₂OCH₂CH₂OH
20 Glycerol monolaurate, C₁₁H₂₃COOCH₂CHOHCH₂OH
Glycerol monooleate, C₁₇H₃₃COOCH₂CHOHCH₂OH
Glycerol monostearate, (C₁₇H₃₅)COOCH₂CHOHCH₂OH

The following examples, all using possible combinations
25 of the necessary components of the invention, are grouped
according to their functional use. It should be understood
that all variations may be used with the appropriate active
ingredient to produce products with different functions and
slightly different characteristics.

30 Air Freshener Emulsion and Aerosol Preparations:Example 1

1.6 grams of methylparaben (0.2% by weight) and
4.0 grams of ethanol (0.5%), were placed in a 2-liter
stainless steel mixing beaker. The two were hand mixed with
35 a spatula until the methylparaben was completely dissolved.

12 grams of Monamid 150 ADY (fatty acid alkanolamide)
(1.5%), 8.0 grams of glycerol (1%), 0.8 grams of Neodol 1,
(1-undecanol, 0.1%), and 2.4 grams of IFF fragrance 6673-AP
(0.3%) were placed in the mixing container. The contents of

the container were hand mixed with a spatula to produce a homogeneous mix.

699.2 grams of deionized water (87.4%) was next placed in the container. Agitation with a Gifford-Wood, Model 1L, homogenizer mixer was initiated and medium shear utilized to the point (5 minutes) of producing a homogeneous, thickened liquid, ringing gel. Lamellar layers were present in the batch at this point and continued to form as the batch stood for an additional couple of hours.

10 Lamellar or liquid crystal structure was indicated by polarized light microscopy, fluorescent probe analysis, and Frequency Response Analysis (FRA).

205.6 grams of the above lamellar system (91%) was placed in a 400 ml. beaker, which was then subjected to sonification for two minutes using a Sonics & Materials, Inc., 600 Watt High Intensity Ultrasonic Processor. Ultrasonic agitation converted the batch to a semi-clear emulsion solution with a viscosity similar to that of water, a pH of 8.4, and a specific gravity of 0.9909. Fluorescent probe analysis and FRA indicated the presence of vesicles.

Example 2

A formulation made up according to the procedure of Example 1 with 3% Monamid 150 ADY, 0.1% Neodol 1, 1% glycerol, and 0.3% fragrance was - before sonification - photographed by TEM as described before.

Figure 1 (40,300 magnification) shows the multilamellar liposome structures present in the lamellar phase of this formulation.

Figure 2 (62,000 magnification), taken after sonification of the lamellar phase, shows the presence of many vesicular structures of an average size of 20-100 nm in the emulsion. Some larger unilamellar vesicles are also present.

Preparation of the pressurized air freshener containing aerosol container:

205.6 grams (91%) of the intermediate described above was placed in a 305 cc. metal can, and a standard dip tube, push-activated vapor tap aerosol valve (Precision Valve Corp. Stem 0.024", vapor tap 0.013") was crimped on. The can was

evacuated to 20 inches (50.8 cm) of vacuum. The can was then pressure filled with 36 ml. (20.2 grams, 9%) of a propellant blend consisting of 33% A-108 propane and 67% of A-17 butane. The aerosol can was held at 130°F for 20 minutes in a hot tank, and the pressure was checked with a hand held pressure gauge (Liquid Filled, U.S. Gauge, 0-160 psi) and found to be 120 psi. The can was then cooled to 72°F (22.2°C). The finished product was found to have a pressure of 54 psi (3796.74 gr/sq. cm).

10 Example 3

A formulation made up according to the procedure of Example 1 but using 1.5% Monamid 150 ADY, 0.1% Neodol 1, 1% glycerol, 0.3% fragrance, 88.10% distilled water and 9% of the propane-butane blend produced a stable, semi-clear liquid phase aerosol system having a pressure of 56 psi (3937.36 gr/sq. cm), while increasing the Monamid level to 6.5% produced an aerosol system having a pressure of 55 psi (3867 gr/sq. cm). Increasing the level of glycerol to 6% produced a system with a pressure of 53 psi (3867 gr/sq. cm).

20 Example 4

A formulation made up according to the procedure of Example 1 but using 2% Monamid 150 ADY, 0.2% methylparaben, 0.5% ethanol, 1% glycerol, 0.3% fragrance, 90% deionized water, 3% n-pentane, and 3% propane produced an aerosol system with a pressure of 55 psi (3867 gr/sq. cm).

25 Example 5

A formulation similar to that of Example 3 but with 6% n-pentane and 3% propane (and 87% water) produced a system with a pressure of 37 psi (2601.47 gr/sq. cm), lower than the ideal pressure for aerosols intended to produce sprays but appropriate for aerosols intended to deliver such products as post-delivery foaming gels.

30 Example 6

As discussed before, the non-ionic surfactant of the aqueous component need not be of a single type. It can be a combination of two types of non-ionic surfactants that interact to produce the vesicular structure of the system.

A formulation made up according to the procedure of Example 1 but using 1.5% Span 80 (sorbitan monooleate), 0.3%

Tween 80 (polyoxyethylene (2) oleyl ether), 0.25% Neodol 1, 1% glycerol, 0.3% fragrance, 88.35% distilled water, and 9% of the propane-butane blend produced an aerosol system having a pressure of 54 psi (3796.74 gr/sq. cm).

5 Example 7

A formulation made up according to the procedure of Example 1 but using 2.5% Monamid 150 ADY, 0.2% methylparaben, 0.1% Neodol 1, 1% ethylene glycol, 0.3% fragrance, 87.1% deionized water, and 9% of the propane-butane blend, produced
10 an aerosol system with a pressure of 55 psi (3867. gr/sq. cm).

Example 8

A formulation made up according to the procedure of Example 1 but using 2.5% Monamid 150 ADY, 0.1% Neodol 1,
15 1% glycerol monooleate, 0.3% fragrance, 87.1% distilled water and 9% of the propane-butane blend produced a stable, semi-clear liquid phase aerosol system having a pressure of 53 psi (3726.43 gr/sq. cm).

Insecticide Emulsion and Aerosol Preparation:

20 Example 9

1.6 grams of methylparaben (0.2%) and 4.0 grams of ethanol (0.5%) were placed in a 2-liter stainless steel mixing beaker. The two were hand mixed with a spatula until the methylparaben was completely dissolved.

25 16 grams of Monamid 150 ADY (2%), 12.0 grams of glycerol (1.5%), and 1.6 grams of 2,2,4-trimethyl pentane (0.2%) were next placed in the mixing container. The contents of the container were hand mixed with a spatula to produce a homogeneous mix.

30 668.8 grams of deionized water (83.6%) was next placed in the container. Agitation with a Gifford-Wood, Model 1L, Homogenizer mixer was initiated and medium shear utilized to the point (5 minutes) of producing a homogeneous, thickened liquid, ringing gel. Lamellar layers were present in the
35 batch at this point and continued to form as the batch stood for an additional couple of hours.

Lamellar, liquid crystal structure was indicated by polarized light microscopy, fluorescent probe analysis, and FRA.

8 grams of pyrethrum extract (Aerosol grade 20% Pyrethrins) (3%) were placed in the container, and the gel was then sheared for 5 minutes to form a homogeneous, milky white, lamellar system.

- 5 207.4 grams (91%) of the above lamellar system was placed in a 400 ml. beaker, which was then subjected to sonification for two minutes using a Sonics & Materials, Inc., 600 Watt (2047.74 BTU/Hr.) High Intensity Ultrasonic Processor. Ultrasonication converted the batch to a milky
10 white, emulsion solution with a viscosity similar to water, pH of 8.94, and a specific gravity of 1.0005. Fluorescent probe analysis and FRA indicated the presence of vesicles.

Preparation of the Pressurized Insecticide-Containing Aerosol Container:

- 15 207.4 grams (91%) of the intermediate described above was placed in a 305 cc. metal can, and a vapor tap aerosol valve was crimped on. The can was evacuated to 20 inches of vacuum. The can was then pressure filled with 36 ml. (20.2g, 9%) of a propellant blend consisting of 33% A-108 propane and
20 67% of A-17 butane. The aerosol can was held at 130°F. (54°C) for 20 minutes in a hot tank, and the pressure was checked with a hand held pressure gauge (Liquid Filled, U.S. Gauge 0-160 psi) and found to be 122 psi (8577.82 gr/sq. cm). The can was then cooled to 72°F. (22.2°C). The finished
25 product was found to have a pressure of 55 psi (3867 gr/sq. cm).

Insect Repellent Emulsion and Aerosol Preparation

Example 10

- 20 grams of Monamid B-442 (linoleamide) (2.5%), 12.0
30 grams of glycerol (1.5%) and 2.4 grams of 2,2,4-trimethyl pentane (0.3%) were next placed in the mixing container. The contents of the container were hand mixed with a spatula to produce a homogeneous mix.

- 66.8 grams of deionized water (71.7%) was next placed in
35 the container. Agitation with a Gifford-Wood, Model 1L, Homogenizer mixer was initiated and medium shear utilized to the point (5 minutes) of producing a homogeneous, thickened liquid, ringing gel. Lamellar layers were present in the

batch at this point and continued to form as the batch stood for an additional couple of hours.

Liquid crystal structure was confirmed by polarized light microscopy, fluorescent probe analysis and FRA.

5 120 grams of DEET (15%) were placed in the container, and the gel was then sheared for 5 minutes to form a homogeneous, milky white, lamellar system.

728 grams of the above batch was placed in a 400 ml. beaker, which was then subjected to sonification for two
10 minutes using a Sonics & Materials, Inc., 600 Watt High Intensity Ultrasonic Processor. Ultrasonication converted the batch to a milky white, vesicular solution with a viscosity similar to water, pH of 8.94, and a specific gravity of 1.0005. Fluorescent probe analysis and FRA
15 indicated the presence of vesicles.

Preparation of the Pressurized Insect Repellent-Containing Aerosol Container:

728 grams of the intermediate described above was placed in a 305 cc. metal can, and a vapor tap aerosol valve was
20 crimped on. The can was evacuated to 20 inches (50.8 cm) of vacuum. The can was then pressure filled with 36 ml. of a propellant blend consisting of 33% A-108 propane and 67% of A-17 butane. The aerosol can was held at 130°F. (54°C) for 20 minutes in a hot tank, and the pressure was checked with a
25 hand held pressure gauge (Liquid Filled, U.S. Gauge 0-160 psi) and found to be 122 psi (8577.82 gr/sq. cm). The can was then cooled to 72°F. (22.2°C). The finished product was found to have a pressure of 55 psi (3867 gr/sq. cm).

Dye-Containing Emulsion Preparation

30 Example 11

A formulation was made up according to the procedure of Example 8 but using 2.5% Monamid 150 ADY, 0.1% Neodol 1, 1.0% glycerol, 0.3% 2,2,4-trimethyl pentane, and 0.02% 5(6) carboxy fluorescein. Capture volumes (CV, defined as the
35 captured volume per gram of surfactant, the surfactant in this case being considered as including both the primary alcohol and the other surfactants) were determined using a dialysis technique (using the carboxy fluorescein as the tracer). The results were: CV for the liquid crystal

system, 1.9 ml/g; CV for the vesicle system, 18.9 ml/g. Thus, the vesicular system is able to entrap and hold a large volume of active organic ingredient.

Figure 3, (46,500 magnification) shows this preparation
5 after sonification. The vesicular structures visible range from 25 to 300 nm, with most in the 20 to 100 nm range.

Other modifications of the aqueous emulsion and of the aerosol delivery system of the present invention will become apparent to those skilled in the art from an examination of
10 the above patent Specification and drawings. Therefore, other variations of the present invention may be made which fall within the scope of the following claims even though such variations were not specifically discussed above.

Industrial Applicability

15 Both the aqueous emulsion preparation and the aerosol delivery system of the present invention can be used to deliver many types of products. Active ingredients to be delivered by such systems can include such things as pesticides, insect repellents, fragrances, emollients,
20 polymers, and polishing or cleansing compounds, etc.

What I claim is:

1. An aqueous emulsion system characterized by the presence of vesicular structures of an average size of 10-300 nm comprising a mixture of a non-ionic surfactant, a C₂-C₁₈
5 primary alcohol, a compound selected from the group consisting of polyhydroxy alcohols, polyhydroxy alcohol esters and mixtures thereof, an active ingredient and water.
2. The aqueous emulsion system of Claim 1 wherein the mixture comprises a non-ionic surfactant, a C₉-C₁₈ primary
10 alcohol, a compound selected from the group consisting of a C₂-C₆ polyhydroxy alcohol, a C₁₀-C₃₀ polyhydroxy alcohol ester and mixtures thereof, an active ingredient and water.
3. The aqueous emulsion system of Claim 1 wherein the mixture comprises 0.25% to 6.5% of a non-ionic surfactant,
15 0.001% to 3.5% of a C₉-C₁₈ primary alcohol, 0.1% to 6% of a C₂-C₆ polyhydroxy alcohol, and 0.01% to 20% of an organic active ingredient, the balance of the emulsion system being water.
4. The aqueous emulsion system of Claim 1 wherein the non-
20 ionic surfactant is a mixture of two or more non-ionic surfactants.
5. The aqueous emulsion system of Claim 1 wherein the non-ionic surfactant is a non-ionic surfactant having a pair of hydrocarbon chains attached to its functional group.
- 25 6. The aqueous emulsion system of Claim 1 wherein the non-ionic surfactant is a non-ionic surfactant having a hydrocarbon chain attached to its functional group.
7. The aqueous emulsion system of Claim 1 wherein the non-ionic surfactant is selected from the group consisting of an
30 ethylene oxide adduct of nonyl phenol, a fatty acid alkanolamide, and an ethoxylated amine.
8. The aqueous emulsion system of Claim 1 wherein the primary alcohol is selected from the group consisting of a C₁₀ alcohol or a C₁₁ alcohol.
- 35 9. The aqueous emulsion system of Claim 1 additionally comprising from 0.1% to 0.5% of a preservative.
10. The aqueous emulsion system of Claim 1 wherein the mixture comprises 0.25% to 6.5% of a fatty acid alkanolamide surfactant, 0.001% to 3.5% of a C₁₁ primary alcohol, 0.1% to

6% of glycerol and 0.01% to 20% of an organic active ingredient, the balance of the emulsion being water.

11. The aqueous emulsion system of Claim 10 wherein the organic active ingredient is an insect repellent.

5 12. The aqueous emulsion system of Claim 10 wherein the organic active ingredient is an air freshening preparation.

13. The aqueous emulsion system of Claim 10 wherein the organic active ingredient is a cleaning and polishing composition.

10 14. The aqueous emulsion system of Claim 10 wherein the organic active ingredient is a dermal treatment material.

15. A method of preparing an aqueous emulsion system characterized by the presence of vesicular structures of an average size of 10-300 nm comprising:

15 placing into a container a non-ionic surfactant,
 a C₂-C₁₈ primary alcohol,
 a compound selected from the group consisting of
polyhydroxy alcohols, polyhydroxy alcohol esters and mixtures
thereof, and

20 an organic active ingredient,
 mixing these ingredients in the container to produce a
homogeneous mixture,
 adding water to the container,
 further mixing the ingredients in the container to form

25 the lamellar stage, and
 adding energy, by means of utilizing a method selected
from the group consisting of a high energy shearing and
sonification, to the lamellar stage to produce the vesicle
containing emulsion.

30 16. The method of preparing an aqueous emulsion system of
Claim 15 wherein the method comprises:

 placing into a container a non-ionic surfactant,
 a C₉-C₁₈ primary alcohol,
 a compound selected from the group consisting of a C₂-C₆
35 polyhydroxy alcohol, a C₁₀-C₃₀ polyhydroxy alcohol ester and
mixtures thereof, and
 an organic active ingredient,
 mixing these ingredients in the container to produce a
homogeneous mixture,

adding water to the container,
further mixing the ingredients in the container to form
the lamellar stage, and

adding energy, by means of utilizing a method selected
5 from the group consisting of a high energy shearing or
sonification, to the lamellar stage to produce the vesicle
containing emulsion.

17. The method of preparing an aqueous emulsion system of
Claim 15 wherein the method comprises:

10 placing into a container from 0.25 to 6.5% by weight of
a non-ionic surfactant,

from 0.001% to 3.5% by weight of a C₉-C₁₈ primary
alcohol,

from 0.1% to 6% of a C₂-C₆ polyhydroxy alcohol, and
15 from 0.01% to 20% of an organic active ingredient,
mixing these ingredients in the container to produce a
homogeneous mixture,

adding water to the container to bring the total weight
to 100%,

20 further mixing the ingredients in the container to form
the lamellar stage, and

adding energy, by means of utilizing a method selected
from the group consisting of a high energy shearing or
sonification, to the emulsified stage to produce the vesicle
25 containing emulsion.

18. The method of preparing an aqueous emulsion system of
Claim 15 wherein the non-ionic surfactant is a mixture of two
or more non-ionic surfactants.

19. The method of preparing an aqueous emulsion system of
30 Claim 15 wherein the non-ionic surfactant is a non-ionic
surfactant having a pair of hydrocarbon chains attached to
its functional group.

20. The method of preparing an aqueous emulsion system of
Claim 15 wherein the non-ionic surfactant is a non-ionic
35 surfactant having a hydrocarbon chain attached to its
functional group.

21. The method of preparing an aqueous emulsion system of
Claim 15 wherein the non-ionic surfactant is a mixture of a
non-ionic surfactant having a pair of hydrocarbon chains

attached to its functional group and a non-ionic surfactant having a hydrocarbon chain attached to its functional group.

22. The method of preparing an aqueous emulsion system of Claim 15 wherein the non-ionic surfactant is selected from the group consisting of an ethylene oxide adduct of nonyl phenol, a fatty acid alkanolamide, and an ethoxylated amine.

23. The method of preparing an aqueous emulsion system of Claim 15 wherein the primary alcohol is selected from the group consisting of a C₁₀ alcohol or a C₁₁ alcohol.

24. The method of preparing an aqueous emulsion system of Claim 15 wherein the aqueous emulsion additionally comprises from 0.1% to 0.5% of a preservative.

25. A method of preparing an aqueous emulsion system characterized by the presence of vesicles comprising:

15 preparing a lamellar phase system by:

placing into a container from 0.25 to 6.5% by weight of a fatty acid alkanolamide surfactant,

from 0.001% to 3.5% by weight of a C₁₁ primary alcohol,

from 0.1% to 6% of glycerol, and

20 from 0.01% to 20% of an organic active ingredient,

mixing these ingredients in the container to produce a homogeneous mixture;

adding water to the container to bring the total weight to 100%,

25 further mixing the ingredients in the container to form a lamellar stage, and

adding energy, by means of utilizing a method selected from the group consisting of a high energy shearing or sonification, to the lamellar stage to produce the vesicle

30 containing emulsion.

26. The method of preparing an aqueous emulsion system of Claim 15 wherein the organic active ingredient is an insect repellent preparation.

27. The method of preparing an aqueous emulsion system of Claim 15 wherein the organic active ingredient is an air freshening preparation.

28. The method of preparing an aqueous emulsion system of Claim 15 wherein the organic active ingredient is a cleaning and polishing preparation.

29. The method of preparing an aqueous emulsion system of Claim 15 wherein the organic active ingredient is a dermal treatment material.

30. An improved two-phase system for delivering an aerosol
5 from a pressurized container characterized by a propellant
reservoiring effect and comprising from 75% to 98% by weight
of an aqueous component and from 25% to 2% by weight of a
propellant component, the aqueous component comprising a
mixture of a non-ionic surfactant, a C₂-C₁₈ primary alcohol,
10 a compound selected from the group consisting of polyhydroxy
alcohols, polyhydroxy alcohol esters and mixtures thereof,
and an active ingredient, the balance of the aqueous
component being water, the propellant component comprising a
C₃-C₅ linear hydrocarbon.

31. The improved two-phase system for delivering an aerosol
15 from a pressurized container of Claim 30 wherein the system
comprises from 75% to 98% by weight of an aqueous component
and from 25% to 2% by weight of a propellant component, the
aqueous component comprising a mixture of a non-ionic
20 surfactant, a C₉-C₁₈ primary alcohol, a compound selected
from the group consisting of a C₂-C₆ polyhydroxy alcohol, a
C₁₀-C₃₀ polyhydroxy alcohol ester and mixtures thereof, and
an active ingredient, the balance of the aqueous component
being water, the propellant component comprising a C₃-C₅
25 linear hydrocarbon.

32. The improved two-phase system for delivering an aerosol
from a pressurized container of Claim 30 wherein the system
comprises from 75% to 98% by weight of an aqueous component
and from 25% to 2% by weight of a propellant component, the
30 aqueous component comprising a mixture of 0.25% to 6.5% of a
non-ionic surfactant, 0.001% to 3.5% of a C₉-C₁₈ primary
alcohol, 0.1% to 6% of a C₂-C₆ polyhydroxy alcohol, and 0.01%
to 20% of an organic active ingredient, the balance of the
aqueous component being water, the propellant component
35 comprising a C₃-C₅ linear hydrocarbon.

33. The system for delivering an aerosol of Claim 30 wherein
the aqueous component comprises from 80% to 90% by weight of
the system and the propellant component comprises from 10% to
2% by weight of the system.

34. The system for delivering an aerosol of Claim 30 wherein the non-ionic surfactant is a mixture of two or more non-ionic surfactants.
35. The system for delivering an aerosol of Claim 30 wherein
5 the non-ionic surfactant is a non-ionic surfactant having a pair of hydrocarbon chains attached to its functional group.
36. The system for delivering an aerosol of Claim 30 wherein the primary alcohol is a non-ionic surfactant having a hydrocarbon chain attached to its functional group.
- 10 37. The system for delivering an aerosol of Claim 30 wherein the propellant is a mixture of a non-ionic surfactant having a pair of hydrocarbon chains attached to its functional group and a non-ionic surfactant having a hydrocarbon chain attached to its functional group.
- 15 38. The system for delivering an aerosol of Claim 30 wherein the non-ionic surfactant is selected from the group consisting of an ethylene oxide adduct of nonyl phenol, a fatty acid alkanolamide, and an ethoxylated amine.
39. The system for delivering an aerosol of Claim 30 wherein
20 the primary alcohol is selected from the group consisting of a C₁₀ alcohol or a C₁₁ alcohol.
40. The system for delivering an aerosol of Claim 30 wherein the aqueous component additionally comprises from 0.1% to 0.5% of a preservative.
- 25 41. The improved two-phase system of Claim 30 wherein the aerosol to be delivered from the pressurized container is an insect repellent preparation.
42. The improved two-phase system of Claim 30 wherein the aerosol to be delivered from the pressurized container is an
30 air freshening preparation.
43. The improved two-phase system of Claim 30 wherein the aerosol to be delivered from the pressurized container is a cleaning and polishing composition.
44. An improved two-phase system for delivering an aerosol
35 from a pressurized container characterized by a propellant reservoiring effect and comprising from 75% to 98% by weight of an aqueous component and from 25% to 2% by weight of a propellant component, the aqueous component comprising a mixture of 0.25% to 6.5% of a fatty acid alkanolamide

surfactant, 0.001% to 3.5% of a C₁₁ primary alcohol, 0.1% to 6% of glycerol and 0.01% to 20% of an organic active ingredient, the balance of the aqueous component being water, the propellant component comprising a mixture of propane and
5 butane.

45. A method of preparing an improved two phase system for delivering an aerosol from a pressurized container, the system having an aqueous component which provides a reservoiring effect for the propellant and a propellant
10 component comprising:
 preparing a lamellar stage of the aqueous component by:
 charging a first container with a non-ionic surfactant,
 a C₂-C₁₈ primary alcohol,
 a compound selected from the group consisting of
15 polyhydroxy alcohols, polyhydroxy alcohol esters and mixtures thereof, and
 an organic active ingredient,
 mixing these ingredients in the container to produce a homogeneous mixture,
20 charging the first container with water,
 further mixing the ingredients in the container to form the lamellar stage,
 adding energy, by means of utilizing a method selected from the group consisting of a high energy shearing or
25 sonification, to the lamellar stage to produce an emulsified stage of the aqueous component, and
 charging a second, aerosol, container with between 75% and 98% by weight of the emulsified stage, and pressurizing the second container containing the emulsified stage with
30 between 25% and 2% by weight of a C₃-C₅ linear hydrocarbon.

46. The method of preparing an improved two phase system for delivering an aerosol from a pressurized container, according to Claim 45 wherein the method comprises:
 preparing a vesicular phase system by:
35 charging a first container with a non-ionic surfactant,
 a C₉-C₁₈ primary alcohol,
 a compound selected from the group consisting of a C₂-C₆ polyhydroxy alcohol, a C₁₀-C₃₀ polyhydroxy alcohol ester and mixtures thereof, and

an organic active ingredient,
mixing these ingredients in the container to produce a
homogeneous mixture,
charging the first container with water,
5 further mixing the ingredients in the container to form
a liquid crystal phase system,
adding energy, by means of utilizing a method selected
from the group consisting of a high energy shearing or
sonification, to the liquid crystal phase system to produce a
10 vesicular phase system, and
charging a second, aerosol, container with between 75%
and 98% by weight of the vesicular phase system, and
pressurizing the second container containing the vesicular
phase system with between 25% and 2% by weight of a C₃-C₅
15 linear hydrocarbon.

47. The method of preparing an improved two phase system for
delivering an aerosol from a pressurized container, according
to Claim 45 wherein the method comprises:
preparing a lamellar stage of the aqueous component by:
20 charging a first container with a 0.25 to 6.5% by weight
of a non-ionic surfactant,
from 0.001% to 3.5% by weight of a C₉-C₁₈ primary
alcohol,
from 0.1% to 6% of a C₂-C₆ polyhydroxy alcohol, and
25 from 0.01% to 20% of an organic active ingredient,
mixing these ingredients in the container to produce a
homogeneous mixture;
charging the first container with water to bring the
total weight to 100%,
30 further mixing the ingredients in the container to form
the lamellar stage,
adding energy, by means of utilizing a method selected
from the group consisting of a high energy shearing or
sonification, to the emulsified stage to produce an
35 emulsified stage of the aqueous component, and
charging a second, aerosol, container with between 75%
and 98% by weight of the emulsified stage, and pressurizing
the second container containing the emulsified stage with
between 25% and 2% by weight of a C₃-C₅ linear hydrocarbon.

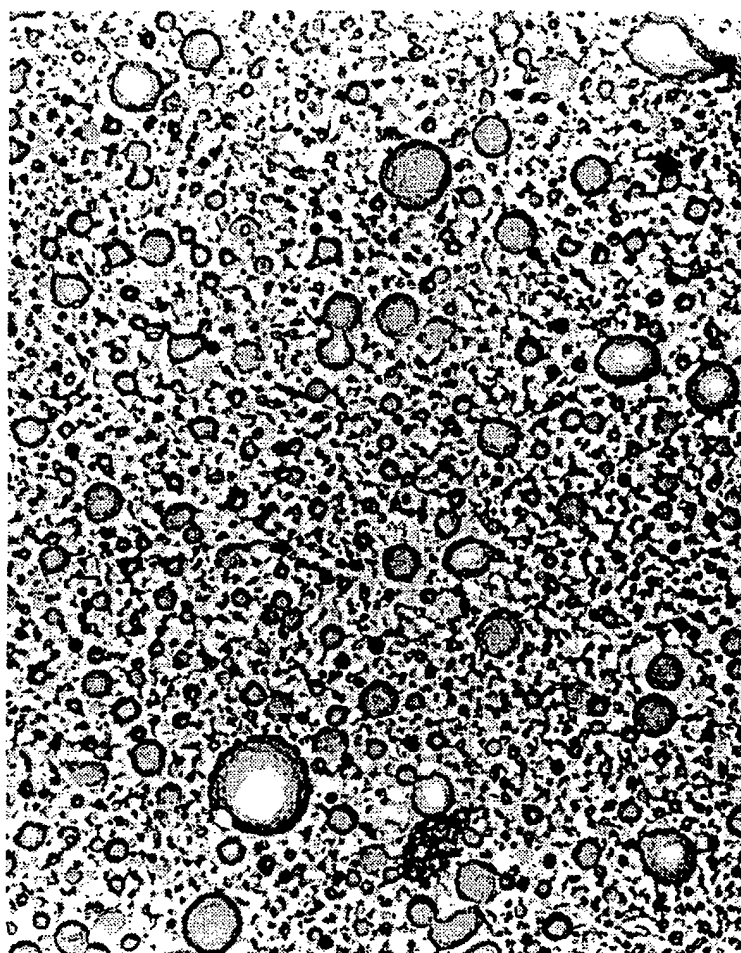
48. The method of preparing an improved two-phase system for delivering an aerosol of Claim 45 wherein the non-ionic surfactant is a mixture of two or more non-ionic surfactants.
49. The method of preparing an improved two-phase system for
5 delivering an aerosol of Claim 45 wherein the non-ionic surfactant is a non-ionic surfactant having a pair of hydrocarbon chains attached to its functional group.
50. The method of preparing an improved two-phase system for delivering an aerosol of Claim 45 wherein the non-ionic
10 surfactant is a non-ionic surfactant having a hydrocarbon chain attached to its functional group.
51. The method of preparing an improved two-phase system for delivering an aerosol of Claim 45 wherein the non-ionic surfactant is a mixture of a non-ionic surfactant having a
15 pair of hydrocarbon chains attached to its functional group and a non-ionic surfactant having a hydrocarbon chain attached to its functional group.
52. The method of preparing an improved two-phase system for delivering an aerosol from a pressurized container of
20 Claim 45 wherein the non-ionic surfactant is selected from the group consisting of an ethylene oxide adduct of nonyl phenol, a fatty acid alkanolamide, and an ethoxylated amine.
53. The method of preparing an improved two-phase system for delivering an aerosol from a pressurized container of
25 Claim 45 wherein the primary alcohol is selected from the group consisting of a C₁₀ alcohol or a C₁₁ alcohol.
54. The method of preparing an improved two-phase system for delivering an aerosol from a pressurized container of
30 Claim 45 wherein the propellant is selected from the group consisting of propane, butane and mixtures thereof.
55. The method of preparing an improved two-phase system for delivering an aerosol from a pressurized container of Claim 45 wherein the aqueous component additionally comprises from 0.1% to 0.5% of a preservative.
- 35 56. The method of preparing an improved two-phase system for delivering an aerosol from a pressurized container of Claim 45 wherein the propellant component is present at concentrations between 10% and 2% by weight.

57. The method of preparing an improved two phase system of Claim 45 wherein the aerosol to be delivered from the pressurized container is an insect repellent preparation.
58. The method of preparing an improved two phase system of Claim 45 wherein the aerosol to be delivered from the pressurized container is an air freshening preparation.
59. The method of preparing an improved two phase system of Claim 45 wherein the aerosol to be delivered from the pressurized container is a cleaning and polishing preparation.
60. A method of preparing an improved two phase system for delivering an aerosol from a pressurized container, the system having an aqueous component and a propellant component, which provide a reservoiring effect for the propellant, comprising:
- preparing a lamellar phase system by:
 - charging a first container with a 0.25 to 6.5% by weight of a fatty acid alkanolamide surfactant,
 - from 0.001% to 3.5% by weight of a C₁₁ primary alcohol,
 - from 0.1% to 6% of glycerol, and
 - from 0.01% to 20% of an organic active ingredient,
 - mixing these ingredients in the container to produce a homogeneous mixture,
 - charging the first container with water to bring the total weight to 100%,
 - further mixing the ingredients in the container to form a lamellar stage,
 - adding energy, by means of utilizing a method selected from the group consisting of a high energy shearing or sonification, to the lamellar stage to produce an emulsified stage of the aqueous component, and
 - charging a second, aerosol, container with between 75% and 98% by weight of the emulsified stage, and pressurizing the second container containing the emulsified stage with between 10% and 2% by weight of a propellant comprising a mixture of propane and butane.



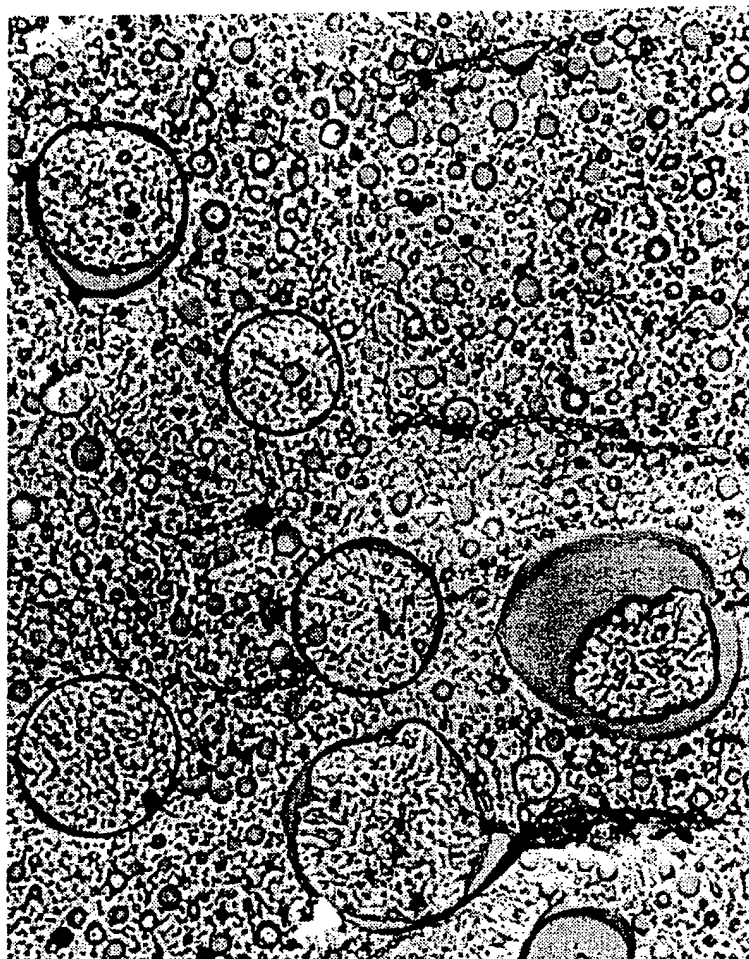
1 μm
100 nm

FIG. 1



└──────────┘ 1 μ m
└──100 nm

FIG. 2



1 μm
100 nm

FIG. 3

INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/06635

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ¹		
According to International Patent Classification (IPC) or to both National Classification and IPC IPC(5): C09K 3/30; B01J 13/00; A61L 9/04; A01N 29/00 US CL : 252/305 254/311/312; 424/45; 71/64.08		
II. FIELDS SEARCHED		
Minimum Documentation Searched ²		
Classification System	Classification Symbols	
US	252/305; 252/311; 252/312 424/45; 71/64.08	
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched ³		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁴		
Category ⁵	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
A	US, A, 4,111,827 Published September, 1978 (Thompson et al.).	1-60
A	US, A, 4,381,066 Published April, 1983 (Page et al.)	1-60
A	US, A, 4,384,661 Published May 1983, (Page et al.)	1-60
X	US, A, 4,439,342 Published March, 1984 (Albanese)	1-60
Y	US, A, 4,860,685 Published August 1989 (Smith)	1-60
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>⁶ Special categories of cited documents: ¹⁴</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>T' later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>X' document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>Y' document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>Z' document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search		Date of Mailing of this International Search Report
08 January 1992		04 FEB 1992
International Searching Authority		Signature of Authorized Officer <i>Nina Bhat</i>
ISA/US		NGUYEN NGOC-BD Fei Nina Bhat INTERNATIONAL DIVISION